

Document made available under the Patent Cooperation Treaty (PCT)

International application number: PCT/NL05/000118

International filing date: 18 February 2005 (18.02.2005)

Document type: Certified copy of priority document

Document details: Country/Office: EP
Number: 04075540.7
Filing date: 19 February 2004 (19.02.2004)

Date of receipt at the International Bureau: 01 April 2005 (01.04.2005)

Remark: Priority document submitted or transmitted to the International Bureau in compliance with Rule 17.1(a) or (b)



World Intellectual Property Organization (WIPO) - Geneva, Switzerland
Organisation Mondiale de la Propriété Intellectuelle (OMPI) - Genève, Suisse

11.03.05



Europäisches
Patentamt

European
Patent Office

Office européen
des brevets

Bescheinigung

Certificate

Attestation

Die angehefteten Unterlagen stimmen mit der ursprünglich eingereichten Fassung der auf dem nächsten Blatt bezeichneten europäischen Patentanmeldung überein.

The attached documents are exact copies of the European patent application described on the following page, as originally filed.

Les documents fixés à cette attestation sont conformes à la version initialement déposée de la demande de brevet européen spécifiée à la page suivante.

Patentanmeldung Nr. Patent application No. Demande de brevet n°

04075540.7

Der Präsident des Europäischen Patentamts;
Im Auftrag

For the President of the European Patent Office

Le Président de l'Office européen des brevets
p.o.

R C van Dijk



Anmeldung Nr:
Application no.: 04075540.7
Demande no:

Anmeldetag:
Date of filing: 19.02.04
Date de dépôt:

Anmelder/Applicant(s)/Demandeur(s):

Nederlandse Organisatie voor toegepast-
natuurwetenschappelijk Onderzoek TNO
Schoemakerstraat 97
2628 VK Delft
PAYS-BAS

Bezeichnung der Erfindung/Title of the invention/Titre de l'invention:
(Falls die Bezeichnung der Erfindung nicht angegeben ist, siehe Beschreibung.
If no title is shown please refer to the description.
Si aucun titre n'est indiqué se referer à la description.)

Process for preparing a particulate material from mammalian hair and use thereof
in paper or paperboard products

In Anspruch genommene Priorität(en) / Priority(ies) claimed /Priorité(s)
revendiquée(s)
Staat/Tag/Aktenzeichen/State/Date/File no./Pays/Date/Numéro de dépôt:

Internationale Patentklassifikation/International Patent Classification/
Classification internationale des brevets:

D01C1/00

Am Anmeldetag benannte Vertragstaaten/Contracting states designated at date of
filing/Etats contractants désignées lors du dépôt:

AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HU IE IT LU MC NL
PT RO SE SI SK TR LI

19. 02. 2004

P67888EP00

(41)

Title: PROCESS FOR PREPARING A PARTICULATE MATERIAL FROM
MAMMALIAN HAIR AND USE THEREOF IN PAPER OR
PAPERBOARD PRODUCTS

The present invention relates to a process for preparing a particulate material from mammalian hair, and the use of the particulate material in paper or paperboard products.

Paper and paperboard products are made of paper pulp consisting of
5 cellulose fibres derived from trees. Since the global demand for paper and
paperboard products is strongly increasing, deforestation is rapidly
accelerating, causing in turn major environmental problems. In order to deal
with the problem of deforestation, in many countries measures have been
taken to ensure that old paper is being recycled and used in the preparation of
10 paper. However, paper has as such the drawback that it can only be recycled a
limited number of times because its intrinsic and required properties
deteriorate as a result of the recycling processes.

Object of the present invention is to provide a cheap and effective
substitute for cellulose fibres, thus ensuring that much less cellulose fibres
15 need to be used in the preparation of paper and paperboard products.

Surprisingly, it has now been found that a very cost-effective and
efficient substitute can be prepared from mammalian hair.

Accordingly, the present invention relates to a process for preparing a
particulate material from mammalian hair which process comprises the
20 following steps:

- (a) subjecting mammalian hair to an oxidation treatment in which the hair is contacted with a solution which comprises a bleaching agent;
- (b) separating the oxidised hair from the solution;
- (c) drying the separated hair; and

(d) subjecting the dried hair to a treatment in which the hair is formed into a particulate material having an average particle size in the range of from 0.5 to 4 mm.

5 The particulate material so obtained can very attractively be used as a substitute for cellulose fibres in the production of paper and paperboard products.

Although various types of mammalian hair can be used in the process of the present invention, the hair of livestock is being preferred, one reason being its readily availability due to today's large, worldwide meat consumption.
10 Another reason is the stiffness of the hair of livestock in combination with excellent mixing properties. Especially, the hair of pigs can very attractively be used in accordance with the present invention.

One single type of mammalian hair can be used or a mixture of various different kinds of mammalian hair can be used. In practice preference will be
15 given to the use of one type of mammalian hair.

In the process according to the invention mammalian hair is subjected in step (a) to an oxidation treatment which is carried out in a solution comprising a bleaching agent. The solution comprises a solution that has been made alkaline (above pH 7) or acidic (below pH 7). Preferably, the solution has
20 been made alkaline by the addition of NaOH, KOH and/or NH₄OH or acidic by the addition of one or more (organic) acid(S). A wide variety of (organic) acids can be used, including for instance acetic acid and formic acid.

The pH value of the alkaline solution to be used in step (a) is preferably in the range of from 9 to 11, more preferably in the range of from 10 to 11, and
25 the pH value of the acidic solution is preferably in the range of 3 to 7, more preferably in the range of from 4 to 6.

Suitable bleaching agents include organic and inorganic peroxides. Preferably, use is made of a bleaching agent selected from the group of hypohalides, perborates, percarbonates, organic peroxides, or hydrogen
30 peroxide. More preferably, the bleaching agent comprises hydrogen peroxide.

One single bleaching or a mixture of different bleaching agents can suitably be applied in the alkaline or acidic solution. In the alkaline solution preferably one or more inorganic peroxides are used, whereas in the acidic solution preferably one or more organic peroxides are used. Suitably, the bleaching agent is used in an amount in the range of from 0.1% (w/w) to 40% (w/w), preferably in the range of from 0.3% (w/w) to 30% (w/w), based on total alkaline or acidic solution.

In step (a) the hair can suitably be contacted with the alkaline or acidic solution over a period of time in the range of from 5 minutes to 16 hours, preferably in the range of from 15 minutes to 10 hours. The temperature to be applied in step (a) is suitably in the range of from room temperature to 100°C, preferably in the range of from 30°C to 80°C.

The hair to be oxidised in step (a) is preferably first subjected to a washing step in which soluble components, such as for instance blood, urine remnants and other animal components, are removed from the hair before the hair is subjected to step (a).

The oxidised hair obtained in step (a) is subsequently separated in step (b) from the alkaline or acidic solution by means of a known technique. For this purpose use can, for instance, be made of a conventional filtering system.

In step (c) the hair which has been separated in step (b) is dried. The drying of the hair can be established by known techniques. The hair can for instance be dried using hot air or washing with a volatile solvent such as ethanol.

In step (d) the dried hair is subjected to a treatment in which hair is formed into a particulate material having an average particle size in the range of from 0.5 to 4 mm. The particulate material can be made by grinding or refining the hair obtained in step (c). Preferably, the treatment in step (d) comprises a refining treatment in which a particulate material is obtained having an average particle size in the range of from 0.5 to 4 mm. Suitably, the

refining treatment can be carried out by standard pulping and mill technology, known to the skilled person.

Preferably, the particulate material has an average particle size in the range of from 1-3 mm, more preferably in the range of from 1.5-2.5 mm. The
5 particulate material preferably comprises fibres.

The particulate material obtained in accordance with the present invention is unique. The present invention therefore also relates to a particulate material prepared from mammalian hair and obtainable by the process according to the present invention.

10 The present invention further relates to a composition comprising the particulate material according to the present invention. Such a composition suitably comprises a paper product wherein the particulate material comprises fibres. The present invention therefore also relates to fibres prepared from mammalian hair in accordance with the present process. The present
15 invention also relates to a paper product comprising fibres in accordance with the present invention. The present invention also relates to paper pulp comprising the particulate material, preferably fibres, according to the present invention.

In the context of the present invention the term "paper product" is
20 meant to include all sorts of papers, such as printing paper, tissue/hygiene, newspaper, office paper, specialties, as well as materials such as cardboard, folding board, box board, undulated board, and 3D board.

The fibres in accordance with the present invention can very attractively be used to substitute cellulose fibres in the preparation of paper products. In a
25 particular embodiment of the present invention the present fibres are used together with old (recycled) paper to produce paper products. The paper products in accordance with the present invention may advantageously comprise the fibres in accordance with the present invention in an amount in the range of from 0.1 to 60 weight %, based on total paper product. Preferably,
30 the paper products in accordance with the present invention comprise the

present fibres in an amount in the range of from 1 to 50 weight %, based on total paper product. More preferably, in an amount in the range of from 3 to 45 weight %, based on total product.

5 The present invention also relates to the use of the particulate material, preferably fibres, according to the present invention in the preparation of a paper product.

Examples

10 Preparation of hair fibres

To a mixture of 250 grams of hair was added 9 liters of water and subsequently the pH of the mixture was brought to a level suitable for oxidation. Then the temperature of the mixture was raised to 65 – 70°C and
15 140 ml of a 30% (w/w) solution of hydrogen peroxide (pH 11) or 30 ml of a 32% (w/w) of per acetic acid (pH 5) was added. The mixture was stirred for 16 hours after which the oxidised hair was isolated by filtration and dried at 70°C. After drying the hair was cut into small fibres with a fibre length between 0.5 and 4 mm using a PFI mill.

20

Evaluation of oxidised hair fibres.

The various samples of oxidised and processed hair fibres (samples of 5, 10, 20 and 30% (w/w)) were mixed with virgin cellulose fibres from eucalyptus in such
25 a way that for each mixture a constant weight was obtained. Also sheets were made using only the virgin Eucalyptus cellulose fibre for comparison and evaluation results were depicted as 0%. The sheets were obtained by using a FRET (Formation and Retention Tester) and applying a vacuum of 0.5 bar. The sheets were dried at 100°C using a Rapid Köthen drying cell. From each
30 mixture three sheets were made.

From each mixture obtained the paper properties were determined.

Volume per mass (cm^3/gram):

- 5 The volume per mass was calculated by dividing the thickness of the sheet by weight per m^2 . Table 1 gives the results of the different sheets.

Table 1. Volume per mass of sheets

% Bleached hair (w/w)	Volume per mass (cm^3/g)
0	1.52
5	1.58
10	1.64
20	1.66
30	1.83

- 10 From the results as depicted in Table 1 the conclusion can be drawn that the volume per mass increases with increased addition of oxidised and processed hair fibres.

Porosity:

- 15 The effect of the addition of oxidised hair fibres on porosity is depicted in Figure 1. From Figure 1 the conclusion can be drawn that the addition of oxidised and processed hair fibres does not have a large influence on the porosity of the formed sheets.

- 20 Short compression test:

The influence of oxidized and processed hair as additive in cellulose pulp on the SCT-Index is depicted in Figure 2. As can be seen from Figure 2 the SCT-

Index properties of the sheets containing processed hair do not differ from the sheet containing only virgin cellulose fibres.

Z-directional tensile:

- 5 The influence of bleached and processed hair on the Z-directional tensile is depicted in Figure 3. Figure 3 shows that also in this case the sheets containing bleached and processed hair fibres do not have an influence on the Z-directional tensile. The hair fibres seem to blend in with the cellulose fibres very well.

10

Tensile index:

- This parameter was measured to evaluate the force at break and gives an indication of the length of the paper needed before it breaks. Figure 4 shows the results when part of the cellulose fiber was replaced by oxidized and
15 processed hair fibres. Figure 4 shows some variation in break length but the conclusion can be drawn that the sheets containing processed hair fibres do not have a negative influence on the performance.

Stretch at break:

- 20 This parameter gives an indication of the amount of stretch of the paper sheet before it breaks. The results are depicted in Figure 5. Figure 5 shows that up to 10% replacement of cellulose fibres by oxidized and processed hair fibres do not give a significant difference in performance, while sheets containing 20 and 30% processed hair fiber do give a higher stretch at break.

25

19. 02. 2004

Claims

(41)

1. A process for preparing a particulate material from mammalian hair which process comprises the following steps:
- (a) subjecting mammalian hair to an oxidation treatment in which the hair is contacted with a solution which comprises a bleaching agent;
- 5 (b) separating the oxidised hair from the solution;
- (c) drying the separated hair; and
- (d) subjecting the dried hair to a treatment in which the hair is formed into a particulate material having an average particle size in the range of from 0.5 to 4 mm.
- 10
2. The process according to claim 1, wherein the particulate material has an average particle size in the range of from 1 to 3 mm.
3. The process according to claim 2, wherein the particulate material has
- 15 an average particle size in the range of from 1.5 to 2.5 mm.
4. The process according to any one of claims 1-3, wherein the bleaching agent is selected from the group consisting of hypohalides, perborates, percarbonates, organic peroxides, or hydrogen peroxide.
- 20
5. The process according to claim 4, wherein the bleaching agent comprises hydrogen peroxide.
6. The process according to any one of the claims 1-5, wherein the
- 25 solution is an alkaline solution having a pH value in the range of from 9 to 11.

7. The process according to any one of claims 1-5, wherein the alkaline solution has a pH value in the range of from 10 to 11.
8. The process according to any or one of the claims 1-5, wherein the solution is an acidic solution having a pH value in the range of from 3 to 7.
9. The process according to claim 8, wherein the acidic solution has a pH value in the range of from 4 to 6.
10. The process according to any one of claims 1-9, wherein the treatment in step (d) is a refining treatment.
11. The process according to any one of claims 1-10, wherein the hair is first subjected to a washing step in which soluble components are removed from the hair before the hair is subjected to step (a).
12. The process according to any one of claims 1-11, wherein the hair is derived from livestock.
13. The process according to claim 12, wherein the hair is derived from pigs.
14. A particulate material derived from mammalian hair and obtainable by a process according to any one of claims 1-13.
15. The particulate material according to claim 14 which comprises fibres.
16. A composition comprising the particulate material according to claim 14 or 15.

17. Paper product comprising the particulate material according to claim 14 or 15.

18. Paper pulp comprising the particulate material according to claim 14 or
5 15.

19. Use of the particulate material according to claim 14 or 15 in the preparation of a paper products.

Abstract

The invention provides a process for preparing a particulate material from mammalian hair which process comprises the following steps:

- (a) subjecting mammalian hair to an oxidation treatment in which the hair is contacted with a solution which comprises a bleaching agent;
- (b) separating the oxidised hair from the solution;
- (c) drying the separated hair; and
- (d) subjecting the dried hair to a treatment in which the hair is formed into a particulate material having an average particle size in the range of from 0.5 to 4 mm.

The invention further relates to the use of the particulate material obtainable by said process and its use in the production of a paper product.

Figure 1

(41)

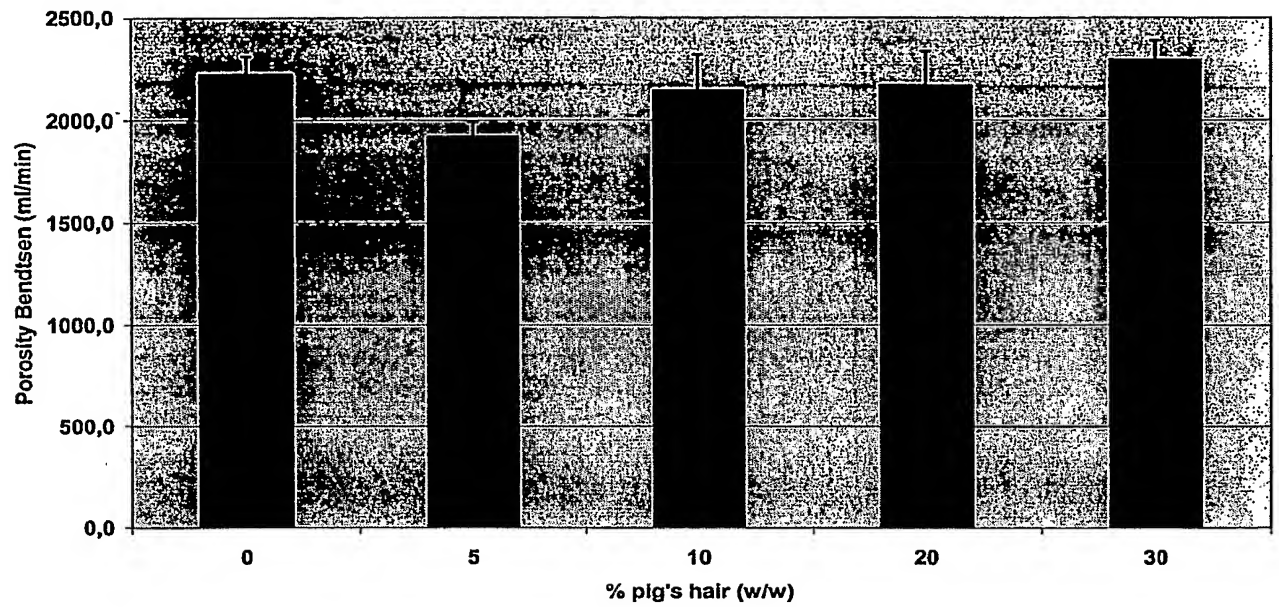


Figure 2

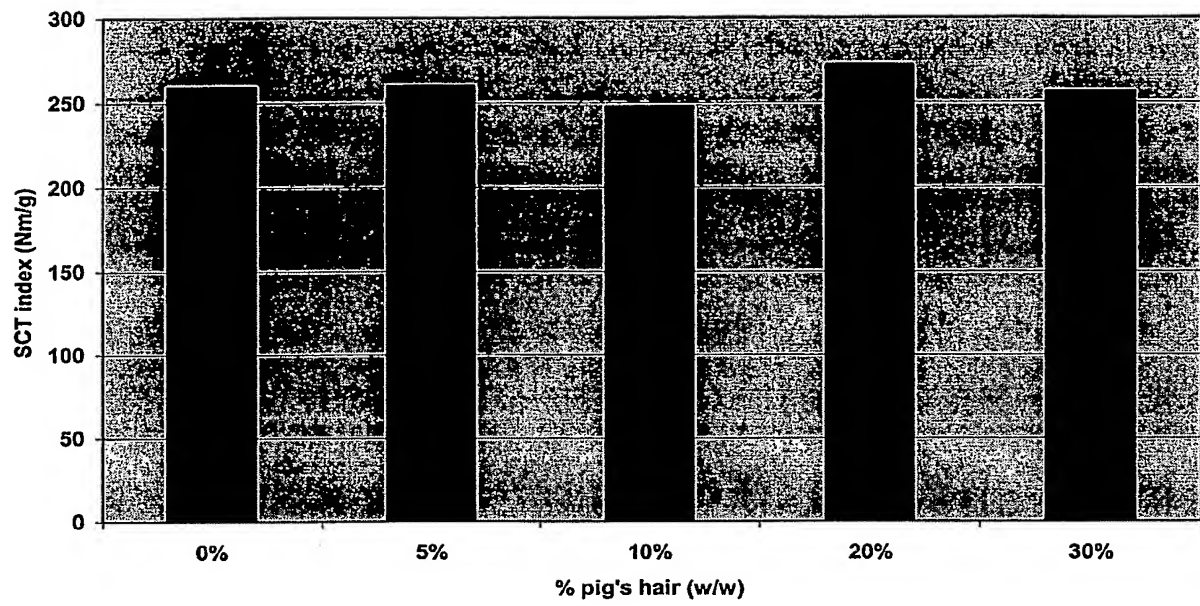


Figure 3

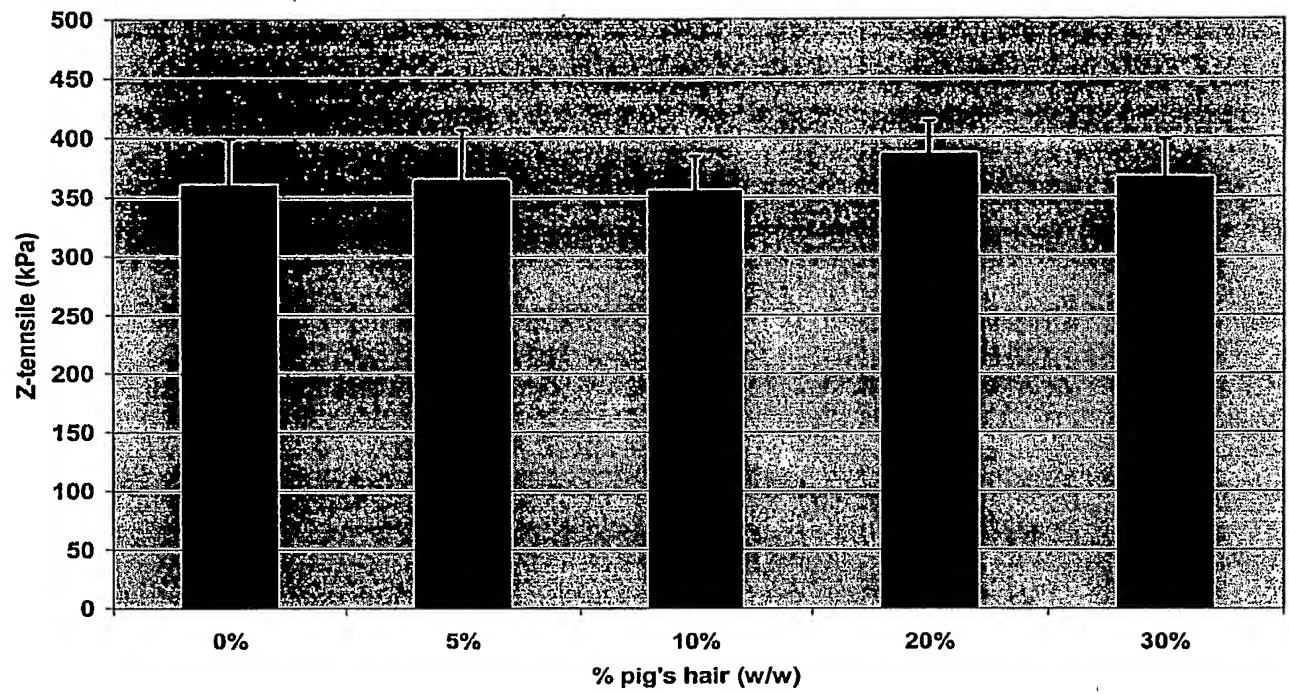


Figure 4

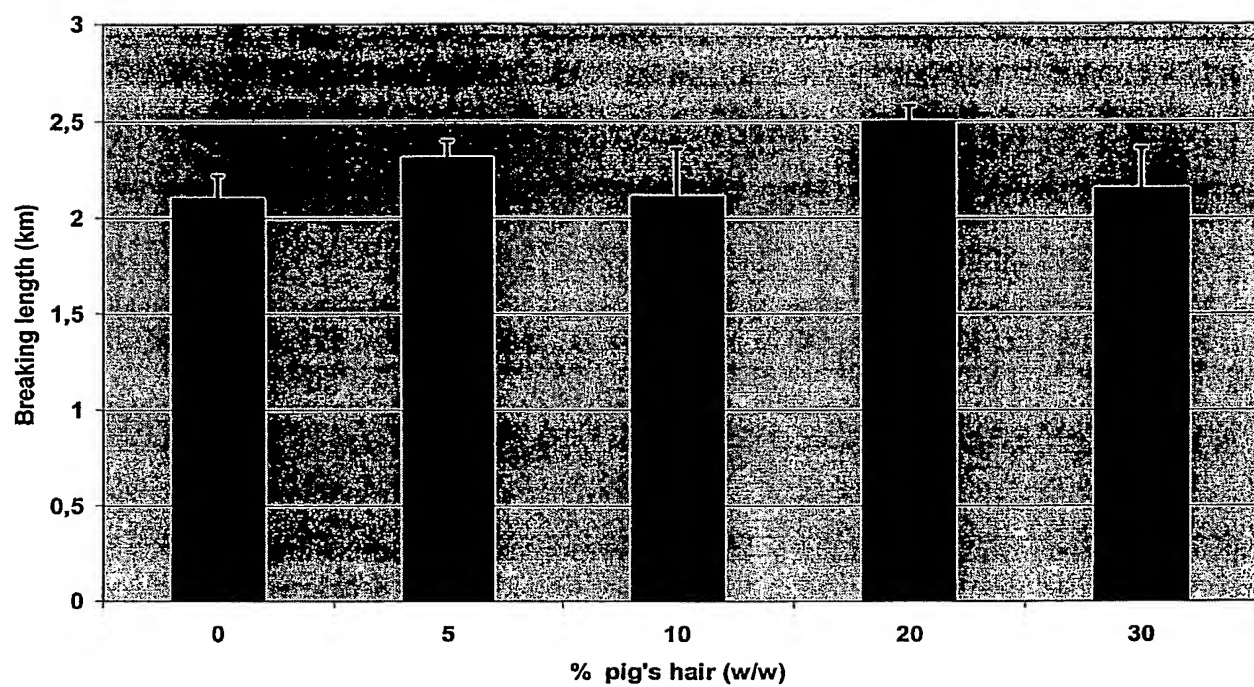


Figure 5

